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With international search report.

(54) Title: PREDICTING LOGARITHMIC REDUCTION VALUES

(57) Abstract

A method of predicting logarithmic reduction values for a membrane filtration system comprising: determining the filtrate flow rate through the membrane, determining the membrane bypass flow rate using integrity test measurements and estimating the logarithmic reduction value using the ratio of determined filtrate flow rate and determined bypass flow rate as (2). Methods of testing the integrity of a porous membrane are also disclosed.

$$LRV = \log_{10}\left(\frac{Q_{filt}}{Q_{Bypass}}\right) \quad (2)$$

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TITLE: PREDICTING LOGARITHMIC REDUCTION VALUES

TECHNICAL FIELD

The present invention relates to a method of predicting logarithmic reduction values in a filtration system and use of such values for control and monitoring of an operating filtration system.

BACKGROUND ART

The ability of a filtration system to remove particles is generally measured in terms of the logarithmic reduction value (LRV). For any given particles, the logarithmic reduction value is defined as:

$$LRV = \log_{10}\left(\frac{C_{\inf}}{C_{eff}}\right) \qquad (1)$$

where C_{inf} = Concentration of particle in the influent

C_{eff} = Concentration of particle in the effluent.

The particle used in the calculation can be any particle of interest, for example, in the case of disinfecting systems it would typically be bacteria or viruses, but may also be suspended solids.

DISCLOSURE OF THE INVENTION

The present invention provides a method of predicting logarithmic reduction values for a membrane filtration system comprising the following steps:

i) determining the filtrate flow rate through the membrane

ii) determining the membrane bypass flow rate using integrity test measurements;

iii) estimating the logarithmic reduction value using the ratio of determined filtrate flow rate and determined bypass flow rate as follows:

$$\mathrm{LRV} = \log_{10}\left(\frac{Q_{filt}}{Q_{Bypass}}\right) \qquad (2)$$

The applicant has developed a number of tests to determine the integrity of filtration membranes, these include the Diffusive Air Flow (DAF) and Pressure Decay Tests (PDT).

According to a further aspect the present invention provides a method of testing the integrity of a porous membrane comprising the steps of;

i) wetting the membrane

ii) applying a gas pressure to one side of the membrane below the bubble point of the membrane pores; and

iii) measuring gas flow across the membrane, said gas flow including diffusive flow through the membrane and flow through leaks and defects in the membrane, said gas flow being related to any defects in the membrane.

Preferably, the gas flow is measured by monitoring the pressure decay of the gas pressure applied to the one side of the membrane. In another preferred form, the gas flow is measured by surrounding the other side of said membrane with a volume of fluid and measuring displacement of said fluid resulting from said gas flow.

Unless the context clearly requires otherwise, throughout the description and the claims, the words ‘comprise’, ‘comprising’, and the like are to be construed in an inclusive as opposed to an exclusive or exhaustive sense; that is to say, in the sense of “including, but not limited to”.

MODES FOR CARRYING OUT THE INVENTION

Preferred examples of these integrity tests will be described, by way of illustration only, as follows. If the lumens of a fully wetted membrane (i.e. all the pores are filled with liquid), are filled with air at a pressure below the bubble point, then the pores of the

membrane will remain wet and there will be no significant air flow through the pores other than a relatively small flow due to diffusion. If a defect is present (such as a broken fibre) then air will flow through the defect, providing of course that the size of the defect is such that it has a bubble point below the test pressure. Therefore, the air flow in such a situation is related directly to the integrity of the membrane system. For an integral system, the air flow will be small and extremely difficult to measure directly. In order to simplify testing and to overcome this problem, the air flow is measured indirectly by measuring liquid flow (in the case of the DAF test) or by measuring pressure decay (in the case of the pressure hold/decay test).

In the Diffusive Air Flow test, the lumens are first pressurised with air to the test pressure (usually 100 kPa) keeping the feed-side of the membrane full. Once the test pressure has been reached the filtrate side is sealed and the feed-side is opened via a measuring valve which can measure liquid flow therethrough. Initially, there is rapid liquid flow through the valve. The high initial flow is predominantly due to the volume of water displaced by membrane expansion and that displaced by water moving to the extremities of the pores of the membrane.

After the initial period, the flow drops to a more stable level and the residual liquid flow is solely due to liquid being displaced by diffusive air flow and air flow through any defects. This air flow is the DAF measurement and is typically volume per unit time.

Diffusive air flow is the flow of air through an integral wetted membrane caused by dissolved air transport through the membrane. The driving force for dissolved air diffusion is the differential partial pressure across the wet membrane. As the solubility of air increases with pressure there is a higher concentration of dissolved air in the liquid layer. The system tends to equalise the concentration in the water layer, which results in a steady-

state air transport across the membrane. At the low pressure side (feed side), the lower partial air pressure allows the air to continuously leave solution. The released air builds up at the top of the feed-side, and there is an accompanying flow of displaced water through the feed side measuring valve.

Defects in the membrane are considered to be "holes" in the membrane that, in effect, penetrate the full width of the walls of the membrane. Air flow through defects is caused by "viscous" gas flow. This means that the air simply flows through a defect, initially replacing the water in the defect. The replacement of water in a defect is relatively easy since defects, by definition, are large compared to the pores and thus have a much lower bubble point (less capillary action). The air flow through defects is related to the size of the defects, and also the number of defects.

Air flow caused by leakage around o-rings is another cause of high flows of air in the DAF measurement.

The DAF measurement is therefore the sum of two components, the diffusive air flow through the membranes (good) and the flow of air through defects in the membranes and o-ring leaks (bad). For any particular filter type, the diffusive air flow through the membranes can be both calculated and measured. By comparing a DAF measurement with the expected value for a fully integral filter, an indication of the relative integrity of the filter can be determined.

In the pressure decay test, as with the DAF test, the lumens are first pressurised with air to the test pressure keeping the feed-side of the membrane full. Once the test pressure has been reached the filtrate side is sealed and the feed-side vented to atmosphere. The drop in pressure of the filtrate system with time is then monitored. This pressure decay

will be directly related to air flow across the membrane and hence system integrity, assuming no leaks elsewhere.

In the DAF test what is measured is the air flow through a membrane. This is normally assumed to be just air flow through defects in the membrane. This air flow though defects can be related to a liquid flow through the same defects, under operating conditions. By comparing the liquid flow through defects and the liquid flow through membrane during filtration, an accurate prediction of a logarithmic reduction value can be calculated from the following equation:

$$\mathrm{LRV} = \log_{10}\left(\frac{Q_{\ell,filt}}{Q_{\ell,DAF}}\right) \qquad (3)$$

where:

$Q_{\ell,filt}$ = the liquid flow through the membrane during filtration (and at the DAF test pressure); and

$Q_{\ell,DAF}$ = the equivalent liquid flow through defects under operating conditions calculated from the DAF test measurements.

The equivalent value of liquid flow through a defect $Q_{\ell,DAF}$ can be calculated from the measured DAF result, that is the air flow through the defect. A DAF test is conducted at a set pressure, known as the test pressure P_{test}. The downstream side of the membrane is vented to atmosphere, and has an atmospheric pressure P_{atm}. When air travels through the membrane via a defect, it expands since the test pressure is greater than atmospheric pressure encountered on the downstream side. Hence the volume of water displaced from the downstream side during a DAF test reflects the volume of air passing through the membrane at atmospheric pressure.

Given the above, a simple means can be devised for correcting for the pressure differences on either side of the membranes when calculating $Q_{\ell,DAF}$ from $Q_{a,DAF}$ (air flow through the defect). First, from the measured volume air flow through the defect, a mass flow of air through the membrane can be calculated. This air mass flow can then be converted back to a volume flow on the downstream side of the membrane.

A defect is considered as a cylindrical hole through the membrane. This may not strictly be the case, but is a good assumption and allows model calculation of flow through a defect. So long as there is laminar flow and the length of the defect is at least, say, ten times its diameter, the measured volume of air flow through a cylindrical defect ($Q_{v,a,defect}$) can be described by the Hagen-Poiseuille equation:

$$Q_{V,a,defect} = \frac{\pi d^4 (P_{test} - P_{atm})}{128 \eta_a l} \qquad (4)$$

where:

d = the defect diameter;

P_{test} = the test (upstream) pressure;

$P_{atm,}$ = the atmospheric (downstream) pressure during the DAF test;

η_a = the viscosity of air at the temperature of filtration and

l = the thickness of the membrane.

The mass flow of air through the defect can be related by the volume flow air with density correction.

$$Q_{m,a,defect} = \rho Q_{v,a,defect} \qquad (5)$$

where:

ρ = the density of the air in the membrane.

The density of air can be calculated from the ideal gas equation:

$$\rho = \frac{PM}{RT} \tag{6}$$

where:

P = the pressure;

M = the molecular weight of air;

R = the gas constant; and

T = the temperature.

The pressure is a difficult parameter to determine for the membrane situation since there is a constant pressure gradient across the membrane, from P_{test} to P_{atm}. A simple way of dealing with this problem is to use the mean pressure.

$$P = \frac{P_{test} + P_{atm}}{2} \tag{7}$$

From equations 5-8 the mass flow of air through a defect is given by:

$$Q_{m,a,defect} = \frac{\pi d^4 (P^2{}_{test} - P^2{}_{atm})}{256\eta_a l} \frac{M}{RT} \tag{8}$$

Now given that the volume of water displaced by the air is at the downstream side, the volume of air that needs to be considered can be calculated from the air mass flow through the defect at atmospheric (downstream) pressure.

Given that, from the ideal gas equation:

$$P_{atm} = \frac{\rho RT}{M} \tag{9}$$

Combining equations 5, 8 and 9 we find:

$$Q_{V,a,defect} = \frac{\pi d^4 (P^2{}_{test} - P^2{}_{atm})}{256\eta_a l P_{atm}} \tag{10}$$

The Hagen-Poiselle equation can also be used to calculate the liquid flow through a defect. The equation for liquid flow is simply the same as the equation for air flow given by equation 4. Since the liquid used is generally water, which is incompressible, none of the considerations of pressure as needed for the air flow, need to be considered. The volume liquid flow through the same defect as above is given by:

$$Q_{V,I,defect} = \frac{\pi d^4 TMP}{128\eta_l l} \qquad (11)$$

where the TMP is the trans-membrane pressure, i.e. the operating pressure of filtration, which is just equal to the pressure difference across the membrane during filtration.

Comparing equations 10 and 11 we find that liquid volume flow through the defect at operational TMP is given by:

$$Q_{V,I,defect} = Q_{V,a,defect} \frac{\eta a}{\eta l} \frac{2P_{test} TMP}{(P^2{}_{test} - P^2{}_{atm})} \qquad (12)$$

Note that $Q_{V,I,defect}$ can be calculated from experiment since $Q_{V,a,defect}$ is just the measured DAF flow, and the rest of the parameters in equation 12 are either known or easily measured.

The measured $Q_{V,I,defect}$ can be used to calculate LRV values. From equation 3 the LRV is given by:

$$\text{LRV} = \log_{10}\left(\frac{Q_{V,I,filt}}{Q_{V,I,defect}}\right) \qquad (13)$$

where $Q_{V,I,filt}$ is just the flow through the membrane in normal filtration mode. Combining equations 12 and 13 the LRV can then be calculated in the following from:

$$\text{LRV} = \log_{10}\left(\frac{Q_{V,I,filt}\eta_l (P^2{}_{test} - P^2{}_{atm})}{Q_{V,a,defect}\eta_a 2P_{atm} TMP}\right) \qquad (14)$$

Note that all parameters in equation 14 are directly measurable.

The bypass flow may also be determined as follows assuming the air flow measured in the DAF test is due to leakage through defects in the membrane and filter seals:

$$Q_{bypass} = Q_{DAF} \times \frac{2\mu_{air} P_{Filt} P_{Vent}}{\mu_{Filt}(P^2{}_{Test} - P^2{}_{Vent})} \tag{15}$$

where:

Q_{bypass} = Equivalent bypass liquid flow

Q_{DAF} = Bypass airflow as measured using DAF test

μ_{air} = Viscosity of air

μ_{filt} = Viscosity of filtrate fluid (usually water during test)

P_{test} = DAF Test pressure, absolute

$_{Vent}$ = DAF Vent pressure, absolute, usually atmospheric

P_{filt} = Filtration transmembrane pressure

The log reduction value can then be estimated as follows:

$$LRV = \log_{10}\left(\frac{Q_{Filt}}{Q_{DAF}} \times \frac{\mu_{Filt}(P^2_{Test} - P^2_{Vent})}{2\mu_{air} P_{Filt} P_{Vent}}\right) \tag{16}$$

where Q_{Filt} = Filtrate flow rate

As with the DAF Test, it is possible to estimate the log reduction value from the pressure decay results. The only additional information required is the pressurised volume of the filtrate pipework during the pressure decay test for the given system. Bypass flow can be estimated from the following equation:

$$Q_{bypass} = \frac{\Delta P V_{Filt}}{P_{atm} t} \times \frac{2\mu_{air} P_{Filt} P_{Vent}}{\mu_{Filt}(P^2{}_{Test} - P^2{}_{Vent})} \tag{17}$$

where:

ΔP = Pressure decay measured over time t

V_{filt} = Volume of filtrate system under test pressure

P_{atm} = Atmospheric Pressure

μ_{air} = Viscosity of air

μ_{filt} = Viscosity of filtrate fluid (usually water during test)

P_{test} = Test pressure, absolute

P_{Vent} = Vent pressure, absolute, usually atmospheric

P_{filt} = Filtration transmembrane pressure

As with the DAF test logarithmic reduction values can be predicted from the bypass flow rate by comparison with the filtrate flow rate by as follows:-

$$\mathrm{LRV} = \log_{10}\left(Q_{Filt} \times \frac{P_{atm}t}{\Delta P V_{Filt}} \times \frac{\mu_{Filt}(P_{Test}^2 - P_{Vent}^2)}{2\mu_{air} P_{Filt} P_{Vent}} \right) \tag{18}$$

where Q_{Filt} = Filtrate flow rate

The DAF and PDT tests can be automated to provide regular process monitoring of system integrity during operation. Further these tests are highly sensitive and enable system integrity to be directly monitored without the requirement for complex water testing. The predicted logarithmic reduction values can be used to monitor and control system performance and loss of integrity. System control can be provided by comparing desired or required LRV's with the predicted LRV's for a particular system and adjusting system performance in response to such comparison.

It will be appreciated that further embodiments and exemplifications of the invention are possible without departing from the spirit or scope of the invention described.

CLAIMS:

1. A method of predicting logarithmic reduction values for a membrane filtration system comprising the following steps:

i) determining the filtrate flow rate through the membrane

ii) determining the membrane bypass flow rate using integrity test measurements;

iii) estimating the logarithmic reduction value using the ratio of determined filtrate flow rate and determined bypass flow rate as follows:

$$LRV = \log_{10}\left(\frac{Q_{filt}}{Q_{Bypass}}\right).$$

2. A method of testing the integrity of a porous membrane comprising the steps of;

i) wetting the membrane

ii) applying a gas pressure to one side of the membrane below the bubble point of the membrane pores; and

iii) measuring gas flow across the membrane, said gas flow including diffusive flow through the membrane and flow through leaks and defects in the membrane, said gas flow being related to any defects in the membrane.

3. A method according to claim 2 wherein the gas flow is measured by monitoring the pressure decay of the gas pressure applied to the one side of the membrane.

4. A method according to claim 2 wherein the gas flow is measured by surrounding the other side of said membrane with a volume of fluid and measuring displacement of said fluid resulting from said gas flow.

5. A method of predicting logarithmic reduction values for a membrane filtration system according to claim 1 wherein the integrity test measurements are produced according the method defined in any one of claims 2 to 4.

INTERNATIONAL SEARCH REPORT

International Application No.
PCT/AU 98/00387

A.	**CLASSIFICATION OF SUBJECT MATTER**
Int Cl[6]:	B01D 65/10

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC6 B01D 65/10

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
AU: IPC as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
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C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US, A, 5417101 (WEICH) 23 May 1995 See whole document	2-4
A.	WO, A, 9628236 (MEMTEC LIMITED) 19 September 1996	
A.	Derwent Abstract Accession No. 91-276439/38 Class S03, JP, A, 3110445, (FUJI PHOTO FILM KK) 10 May 1991	

☐ Further documents are listed in the continuation of Box C

☒ See patent family annex

* Special categories of cited documents:

"A" Document defining the general state of the art which is not considered to be of particular relevance
"E" earlier document but published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
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"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
27 July 1998	-4 AUG 1998
Name and mailing address of the ISA/AU AUSTRALIAN PATENT OFFICE PO BOX 200 WODEN ACT 2606 AUSTRALIA Facsimile No.: (02) 6285 3929	Authorized officer **M. OLLEY** Telephone No.: (02) 6283 2143

INTERNATIONAL SEARCH REPORT

International Application No.
PCT/AU 98/00387

Box 1 Observations where certain claims were found unsearchable (Continuation of item 1 of first sheet)

This International Search Report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. [X] Claims Nos.: 1,5
   because they relate to subject matter not required to be searched by this Authority, namely:
   performing a purely mental act, ie. Putting two values obtained by any measuring means, into a formula to obtain a third value.

2. [ ] Claims Nos.:
   because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3. [ ] Claims Nos.:
   because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a)

Box II Observations where unity of invention is lacking (Continuation of item 2 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

a) Claim 1 is to a method of predicting logarithmic reduction values.

b) Claims 2-4 are to a method of testing the integrity of a porous membrane.

1. [ ] As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims
2. [X] As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.
3. [ ] As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:
4. [ ] No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

[ ] The additional search fees were accompanied by the applicant's protest.

[ ] No protest accompanied the payment of additional search fees.

International Application No.

PCT/AU 98/00387

INTERNATIONAL SEARCH REPORT
Information on patent family members

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent Document Cited in Search Report		Patent Family Member					
US	5417101	CA	2070110	DE	4119040	EP	518250
WO	9628236	AU	49319/96	CA	2214997	EP	814887

END OF ANNEX